IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of	)	
	)	
Keiji Ishibashi	)	Group 1762
	)	
Serial No. 09/633,002	)	Examiner: Wesley D. Markham
	)	
Filed: August 4, 2000	)	
	)	
Title: A HOT ELEMENT CVD APPARATUS AND A METHOD FOR REMOVING A DEPOSITED FILM	)	

DECLARATION UNDER 37 C.F.R. §1.132

I, Keiji Ishibashi, declare that:

1. I am the inventor of the invention set forth and claimed in U.S. Patent application Serial No. 09/633,002, filed August 4, 2000, and entitled "A HOT ELEMENT CVD APPARATUS AND A METHOD FOR REMOVING A DEPOSITED FILM."

2. On June 21, 1999, I prepared a Japanese language document entitled "Examination of Cat-CVD in-situ cleaning," VH4700-0631, which is attached hereto as Exhibit 1, together with an English translation thereof, Exhibit 2.

3. After preparation of the document, Exhibit 1, it was kept confidential within Anelva Corporation, my employer, and was distributed within the Corporation to a limited number of persons on a confidential basis. The document and the information contained therein were considered a company trade secret by the Corporation.

4. I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true and further that

these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Keiji Ishibashi

Nov. 6. 2003
Date

Attachments - Exhibits 1 and 2

配付先 下記各位殿	Cat-CVD in-situクリーニング法の検討	VH4700-0631 1/3 1999年 6月 21日 発行元 プロセス開発研究所 承認 / 査閲 / 作成 石橋

配付先

MMD	高田B，池田K，野村T
M&TS	櫻井S
営業·研装	松本S
西支社	佐枝K，福永K
プロ開研	細川所長，岡田SD，野上K，田中K，本田T，柄澤T

1．はじめに

P-CVD装置では装置内付着膜のin-situクリーニング法が確立されている。しかし、Cat-CVD装置におけるin-situクリーニング法についてはこれまで提案されていない。

今回、フィラメント温度が高ければクリーニングガスのフィラメント付着時間が反応時間よりも短く、フィラメント自身はエッチングされないと仮定し、通電加熱したフィラメントによりクリーニングガスを分解してin-situクリーニングを行う方法について検討した。

2．実験方法

今回実験に用いた装置はC-9010(Bチャンバ)である。実験条件を以下に示す。

＜条件＞

フィラメント	ϕ0.505×ℓ100 Wワイヤ
通電電流	16A (約2000℃)，22A (約2500℃)
到達圧力	21～25Pa
クリーニングガス	NF_3 (100sccm)
クリーニング圧力	約40Pa

以上の条件にて、先ず真空中で1時間通電のみを行い、その後通電保持した状態で1時間NF_3を導入した場合について、処理前後の線径変化をマイクロメータにより測定した。また、比較のために真空中で2時間通電のみを行った場合の線径変化も測定した。さらに、通電電流22AでNF_3を導入した場合については、フィラメントから約2cmと約15cmの位置にSiウェハを配置し、処理前後のウェハの厚みを同じくマイクロメータで測定し、エッチング速度を求めた。

3．結果

得られた結果を表に示す。

通電電流16A、22Aとも通電のみで線径変化があった。Wの蒸気圧から見積もられる2500℃、2hの線径変化は1μm程度であり、実験結果はこれに比べ遥かに大きかった。この原因は、到達圧力が高かったことから酸化による蒸発と推定される。(到達圧力が高かったのは、メインポンプがないこととリークのためである。)

表　真空中で2時間通電のみを行った場合と、真空中で1時間通電した後に通電した状態で1時間NF_3を導入した場合の線径変化.

通電電流［A］<温度［℃］>	線径変化［μm］<初期→処理後>	
	真空中(2h)	真空中(1h)＋NF_3導入(1h)
16A<約2000>	-20<505→485>	-39<505→466>
22A<約2500>	-30<505→475>	-19<505→486>

通電電流16A、フィラメント温度約2000℃では、通電のみよりもクリーニング処理を行った方が線径変化が大きく、NF_3によりフィラメント自身がエッチングされたことがわかる。これに対し、通電電流22A、フィラメント温度約2500℃では、通電のみよりもクリーニング処理を行った方が線径変化が小さく、NF_3によるフィラメント自身のエッチングは生じていないものと推測される。ただし、いずれの通電電流でもクリーニング処理を行ったフィラメントの両端部(給電端子付近の低温部)では顕著なエッチングが観られた。(いずれの通電電流でもNF_3導入後約1.5hでその部分より断線。)

通電電流22A、フィラメント温度約2500℃でNF_3を導入した場合、フィラメントから約2cm及び約15cmの位置に配置したSiウェハの厚さの変化は各々約10μm及び約2.2μmであった。これらから求めたエッチング速度は、各々約167nm/minと約37nm/minであった。

4．まとめ

今回の実験で次のことが確認され、in-situクリーニングの可能性が示唆された。

・フィラメントを加熱することでクリーニングガス(NF_3)を分解でき、Siのエッチングが可能であること。

・フィラメントは温度が高い(2500℃程度)とエッチングされない傾向があること。

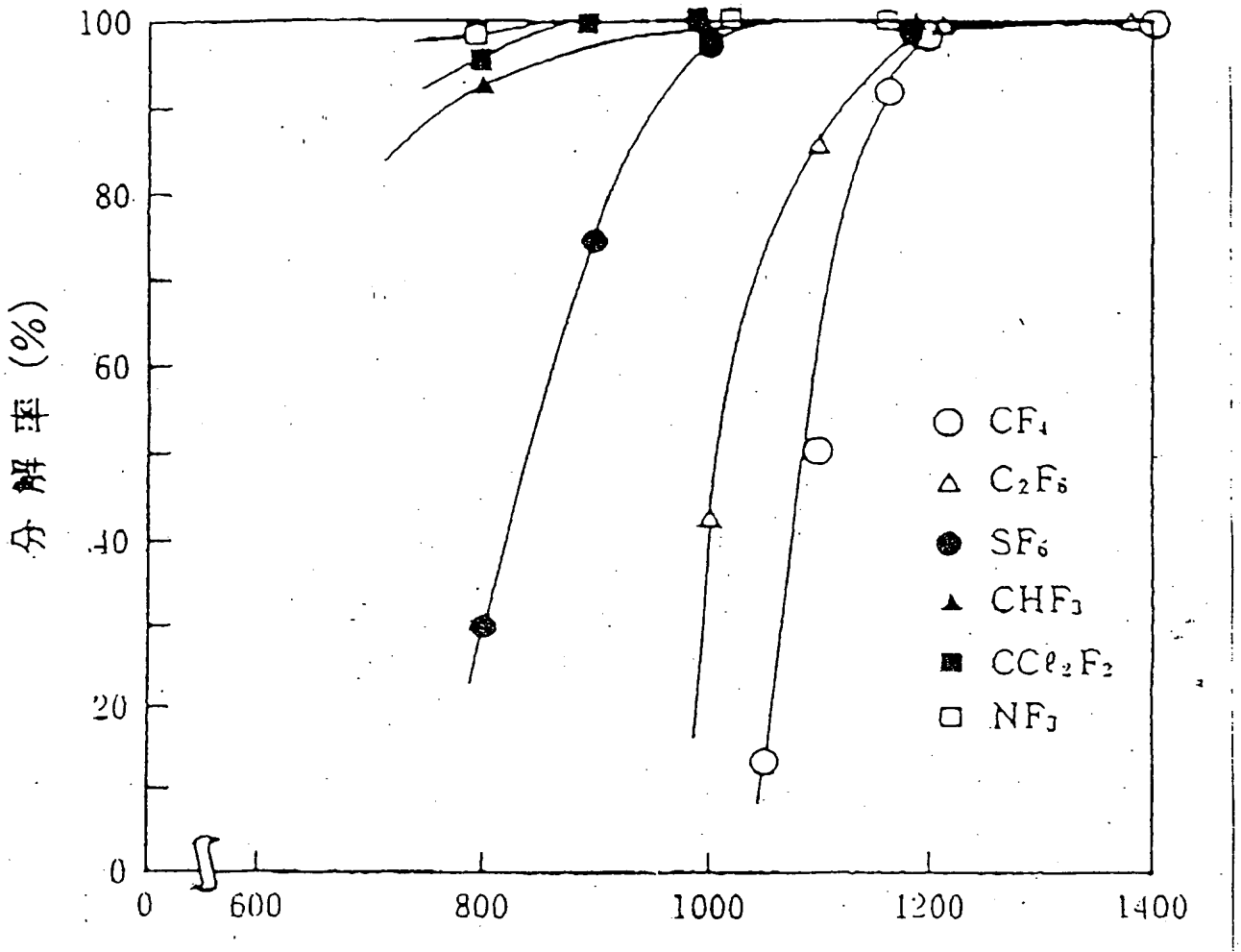
ただし、フィラメント両端部(給電端子付近の低温部)のエッチング対策が必要である。

［所感］

今回の実験はCat-CVD装置におけるin-situクリーニングの可能性を探るための予備評価として行ったもので、リーク箇所の特定とその対策は大がかりとなることを理由に見送った。そのため通電のみでも線径が変化してしまい、通電電流22Aにおいてエッチングされない傾向は観られたものの、エッチングの有無を明確にすることはできなかった。今後、実際の装置でフィラメント両端部(給電端子付近の低温部)のエッチング対策も含め、実証したいと考えている。尚、このフィラメント両端部は成膜時に最も膜堆積を生じるので、この膜がクリーニングされる間にチャンバー内のクリーニングができるのではないかと内心期待をしている。

［付録］

参考として各種フッ素系ガスの温度に対する分解率のグラフ(ウエキコーポレーション大竹氏より提供)を3／3に添付する。



各種フッ素系ガスの分解率

VERIFICATION OF TRANSLATION

I, the below named translator, hereby declare that:

My name and post office address are as stated below; that I am knowledgeable in the Japanese language and in the English language; and that I believe the English translation of the attached Japanese paper is a true and complete translation.

I hereby declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statement may jeopardize the validity of any application based thereon.

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Date: November 7, 2003

Distributed To: Each described below	Examination of Cat-CVD in-situ cleaning	VH4700-0631		1/3
		June 21, 1999		
		Process Dev. Institute		
		Approved by	Inspected by	Prepared by
				Ishibashi

Distributed to:
MMD : Takada B, Ikeda K, Nomura T
M&TS : Sakurai S
Business and Res. Device : Matsumoto S
West branch : Sae K, Fukunaga K
Process Dev.& Res. : Hosokawa director, Okada SD, Nogami K, Tanaka K, Honda T, Karasawa T

1. Introduction

The in-situ cleaning method for removing the films deposited inside the PCVD apparatus is already established. However, the in-situ cleaning method for Cat-CVD apparatus has not been proposed yet.

Then, assuming that the retention time of cleaning gas on the filament becomes shorter at the high temperature and therefore the filament may be prevented from being etched by cleaning gas at a high temperature, I investigated the in-situ cleaning in which a cleaning gas was decomposed with a filament heated by electric current.

2. Experiment method

The apparatus C-9010 (B chamber) was used for experiments. Experiment conditions are shown below.

<Conditions>

Filament :Tungsten (W) wire with a diameter of 0.505mm and a length of 100 mm
Supplied electric current: 16A (about 2000 °C) or 22A (about 2500 °C)
Ultimate vacuum: 21- 25 Pa
Cleaning gas: NF_3 (100 sccm)
Cleaning pressure: about 40 Pa

Under these conditions, firstly, electric current was supplied to the filament in the vacuum for 1 hour, and then NF_3 is introduced for 1 hour at the same temperature. The diameter of wire was measured with a micrometer before and after the processing. For comparison, the wire diameter was also measured before and after the 2-hour supply of electric current in the vacuum. Furthermore, in the case where NF_3 was introduced at the electric current of 22A, a Si wafer was placed 2cm or 15 cm away from the filament. The wafer thickness was also measured with the micrometer before and after processing to obtain the etching rate.

3. Result

The result is shown in the Table. The diameter of wire was reduced even when each of electric currents of 16 A and 22 A was supplied in the vacuum atmosphere without the cleaning gas. The diameter change was much larger than that (about 1 μm) expected from the vapor pressure of W at 2500 °C for 2 hours. This may be caused by the oxidation of filament due to the low ultimate vacuum, increasing the filament evaporation. (This low ultimate vacuum was due to the leakage of chamber and the low-performance exhausting system having no main pump.)

Table: The changes of wire diameter when electric current was supplied to the filament for 2 hours in the vacuum atmosphere, and when the electric current was supplied to the filament in the vacuum atmosphere for 1 hour and thereafter in the NF_3 gas atmosphere for 1 hour.

Electric current [A] <Temperature [℃] >	Wire diameter change [μm] < before and after a processing >	
	In vacuum (2h)	In NF_3 atmosphere (1h) after in vacuum (1h)
16A <about 2000>	-20 <505 and 485>	-39 <505 and 466>
22A <about 2500>	-30 <505 and 475>	-19 <505 and 486>

In the case where the electric current of 16 A was supplied(that is, the filament was heated to about 2000 ℃), the wire diameter was reduced more in the cleaning gas atmosphere than in the vacuum, showing that the filament was etched by NF_3. In contrast, in the case where the electric current of 22A was supplied, the wire diameter was reduced less in the cleaning gas atmosphere than in vacuum, which may mean that the filament was not etched by NF_3 at the filament temperature of 2500 ℃. However, both ends of filament (which were close to feeding terminals and at lower temperature) were observed to be etched in the cleaning gas atmosphere for each case of 16 A and 22 A. (The wires were broken around these portions 1.5 hours after NF_3 was introduced in both cases.)

In addition, when NF_3 was introduced at electric current of 22 A and filament temperature of about 2500 ℃, Si wafers which were placed about 2cm and 15cm away from the filament were reduced by about 10 μm and 2.2 μm in thickness, respectively, corresponding to the etching rates of about 167 nm/min and 37 nm/min, respectively.

4. Conclusion

The followings are derived from the experiment results, suggesting the possibility of in-situ cleaning in Cat-CVD:

- Cleaning gas (NF_3) can be decomposed by heating a filament to etch silicon.
- The filament has tendency not to be etched at a high temperature (of about 2500 ℃).

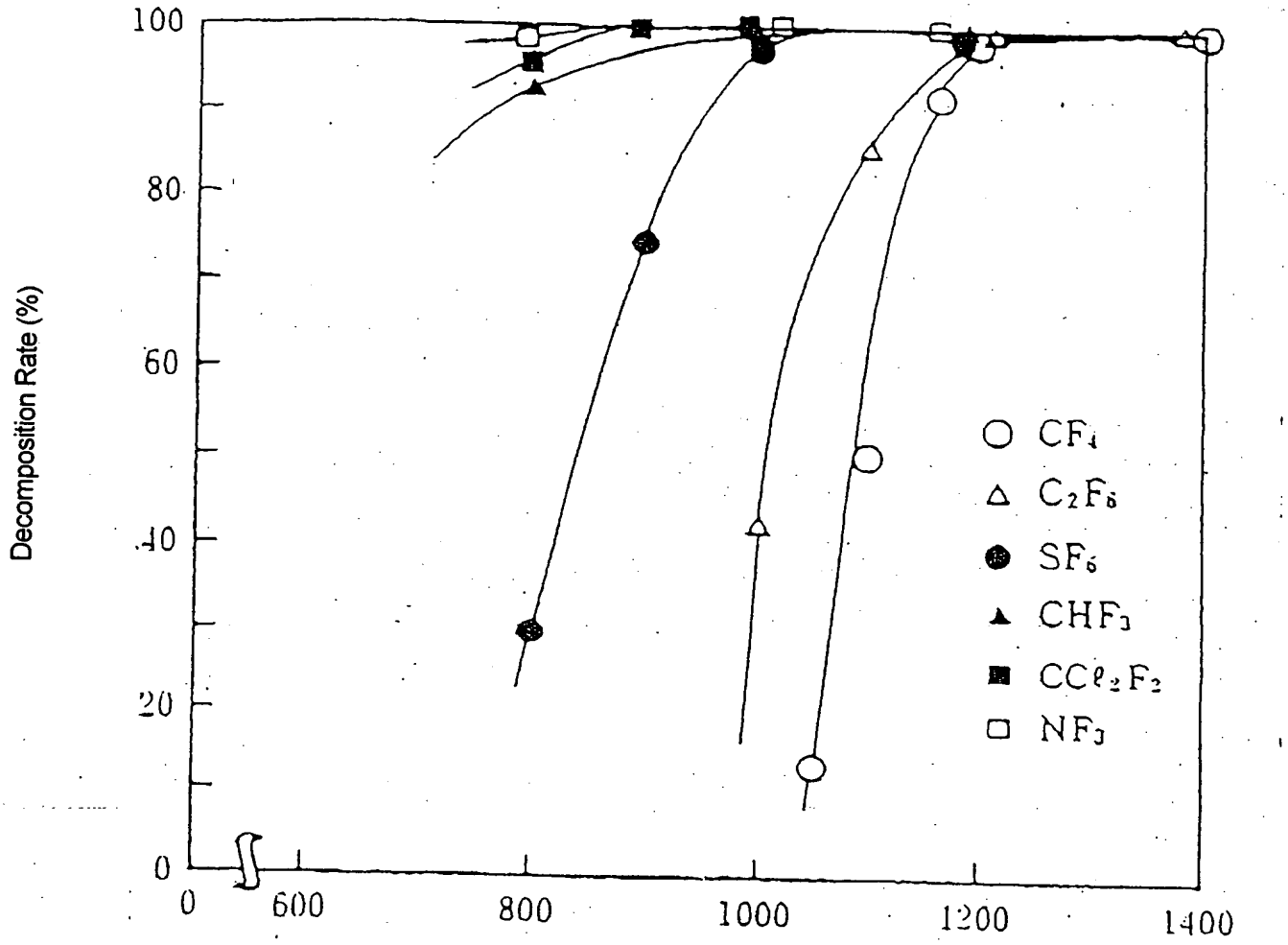
However, there is required the measure to prevent the etching of filament ends (the lower-temperature portions near feeding terminals).

[Impressions]

The above-mentioned experiment was carried out to roughly evaluate the possibility of in-situ cleaning in Cat-CVD apparatus. Thus, I did not try to locate the leak portions or repair the leakage since the tremendous labor seemed to be required. As a result, the wire diameter was reduced even when the electric current was supplied in the vacuum, which made it impossible to clearly make sure if the etching due to cleaning gas would be prevented although the tendency not to be etched at the current of 22 A was observed. Therefore, I am going to prove the etching will be completely prevented at high temperature and find out the measures to prevent the etching of filament at the ends in an actual Cat-CVD apparatus. Notwithstanding, since the film deposits most at the filament ends during film formation, such deposition is expected to prevent the etching of filament ends during the cleaning of chamber.

[Appendix]

The decomposition rates of a variety of fluorine-containing gases are shown as a function of temperature in the graph for reference, which was provided by Mr. Ohtake of UEKI Corporation.



Decomposition Rate of Fluorine-containing gases